

Southall Bros. & Barclay,
(LIMITED)

**TWENTIETH
LABORATORY REPORT**

BIRMINGHAM,

1912.

TWENTIETH ANNUAL REPORT

FROM THE
ANALYTICAL LABORATORIES
OF
SOUTHALL BROS. & BARCLAY
(LIMITED).

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BIRMINGHAM,

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PREFACE.

THE twentieth issue of this record of results from our Laboratory Journal appears at a time when a new edition of the British Pharmacopœia is nearly due.

As will be known by those who have studied our Reports during past years, we have devoted a vast amount of time to advocating the principle of standardisation of galenicals. It has been gratifying to us to notice that by arousing public attention and by the copious illustrations given in our reports, the principle which we have advocated has made great progress, and we are quite hoping that fuller recognition than was the case in the Pharmacopœia of 1898, will be afforded in the new edition.

In this connexion also, we trust that those responsible for the publication of the new Pharmacopœia will avoid the introduction of standards and limits for *materia medica*, which are either very difficult or, in some cases, impracticable to obtain on the manufacturing scale. This has been illustrated in our Reports, and we cannot but think that the yearly accumulation of figures published therein should prove helpful in this respect.

In the present number we bring fresh experimental evidence on that much-discussed preparation, Sweet Spirit of Nitre. Other articles which should prove of special interest are those on the composition of the Oil of Male Fern and of the Potassium Binoxalate of commerce. Indeed it may be said that the whole range of subjects dealt with lies within the borders and interests of scientific pharmacy.

BIRMINGHAM,

January, 1912.

DATA.

Unless otherwise specified, the following data apply to all figures given in the following pages

Specific gravity .. $d_{15.5}^{15.5}$ C.

Optical rotation .. —degree of rotation, with sodium light, using 100 mm. tube.

Temperature in degrees centigrade.

Refractive indices at 15.5 C., unless otherwise stated.

Acid, Ester, Saponification values

are equivalent in each case to the number of milligrammes of KHO required for 1 gramme of the substance, to neutralize the acid, saponify the Ester, or in the case of saponification to the sum of these two.

Iodine absorption figures are all obtained by the method of Wijs.

CRUDE DRUGS, FIXED OILS, WAXES, etc.

THE contents of this and the following sections are taken directly from our Laboratory Journal for 1911, and refer to the examination of drugs, oils, etc., in the condition in which they occur in commerce. We believe that the publication of results of this kind is of interest and also of importance in tending to prevent the fixing of arbitrary standards for crude drugs, which are either unattainable commercially or else can only be secured at a disproportionate cost.

It is to this end that we again include some figures for the ash-yield of powdered drugs, a subject which was dealt with fully in our 11th, 12th, and 13th reports, but which we have not touched upon recently. In this connexion we may say, that unless otherwise stated, the result apply to drugs of good quality ground in our own mills on a manufacturing scale.

Acacia.—The ash-yield from several samples of Acacia after grinding has varied between 2.23 and 2.89 per cent., averaging 2.53.

Aconite Leaves.—Ash of a single sample of the powdered drug proved to be 11.64 per cent.

Almond Oil.—We again record our experience that the index of refraction 1.4729 to 1.4730 laid down by *Bird and Lucas* (*P.J.*, 1910 [2] p. 468) is too high.

Working on eleven samples from perfectly reliable sources we have found this factor to vary between 1·4710 and 1·4720.

Almond Oil (from Peach or Apricot Kernels).—The majority of parcels have proved satisfactory, the figures obtained for seven samples being :—

Specific gravity	0·9180 to	0·9205
Refractive index	1·4710 to	1·4728
Iodine absorbed	...	100·70	to	103·00 per cent.

A sample from a foreign source gave the following figures, indicating sophistication :—

Iodine absorbed	139·60	per cent.
Refractive index	1·4752	

Aloes, Barbados.—The following figures were obtained by the examination of seven specimens of the powdered drug :—

Ash	1·62 to	2·88	per cent.
Cold water soluble	42·9	to	72·3	„

Only one of these reached the 70 per cent. solubility required by the Pharmacopœia.

Aloes, Cape.—A single specimen of the powder yielded 57·0 per cent. to cold water.

Aloes, Socotrine.—Six samples of the crude entire drug yielded from 35·17 to 47·58 per cent. of matter soluble in cold water. Three specimens of the powdered drug showed 51·46, 52·50, 57·70 per cent. respectively.

Araroba.—Two parcels examined proved to contain 51·9 and 50·2 per cent. respectively of substances soluble in chloroform, these results approximating very closely to the official minimum yield of 50 per cent.

Arnica.—A consignment of this drug on examination was found to contain a large proportion, roughly, 75 per cent. of a foreign rhizome, with numerous wiry roots. Mr. E. M. Holmes, who kindly examined this, reported that it was probably the rhizome of a species of *Phlox*. The substitution was quite a crude one, and its detection presented no difficulty.

Asafetida.—A large number of samples have been examined, with the usual widely-varying results. One specimen yielded as little as 1.89 per cent. of ash, while, at the other extreme, 77.16 per cent. was the maximum figure obtained.

With regard to the proportion of matter soluble in alcohol (90 per cent.), we have to record that we have never yet met with a sample of this drug yielding the 65 per cent. laid down by the Pharmacopœia as a minimum. The highest figure obtained during the year was from a sample with but 4.03 per cent. of ash, and which yielded 51.20 per cent. soluble in alcohol (90 per cent.). We note that the United States Pharmacopœia requires 50 per cent. to be soluble in alcohol (about 95 per cent.), a standard which seems to us a more reasonable one.

Beeswax, white.—Twelve samples have been examined, eleven of which gave normal results (*see below*), while the remaining sample proved, according to *Weinwurm's* test, to contain a considerable proportion of paraffin. It is interesting to note that in this instance the mixture was of such a nature as to mask the normal effect of the paraffin wax on the acid and saponification figures. We notice that in the 1910 issue of the *Ph.G.*, the acid number of white beeswax is fixed at 18.7 to 22.4, whereas that of the yellow variety is given as 18.7 to 24.3. Our experience is contrary to this, and we have pointed out

that the white beeswax in this country has almost invariably the higher acid value.

	NORMAL SAMPLES.		ADULTERATED.
Specific gravity ...	0.957 to	0.965	0.937
Melting-point ...	62° to	64°	63°
Acid value ...	21.06 to	24.07	23.60
Saponification value	98.18 to	102.70	101.10

Beeswax, yellow.—Ten samples were subjected to analysis, in one of which paraffin wax was detected. The figures obtained were:—

	NINE NORMAL SAMPLES.		ADULTERATED.
Specific gravity ...	0.958 to	0.967	0.942
Melting-point ...	62° to	64°	59°
Acid value ...	18.47 to	22.18	18.58
Saponification value	90.82 to	96.63	91.87

Belladonna Leaves.—A sample of German leaves of good quality yielded a powder containing 0.63 per cent. of total alkaloids (by titration), and giving 14.19 per cent. of ash.

Belladonna Root.—The only root examined yielded 0.39 per cent. of total alkaloids (by titration).

Cade Oil.—The only sample examined had a specific gravity 1.016, and was free from furfural and catechol.

Canella.—Ash-yield of two samples of the powdered drug, 6.23 and 7.78 per cent. respectively.

Cannabis Indica.—Four samples of Indian-grown drug yielded:—

Soluble in alcohol (90 per cent.)				
	12.7 to	15.2 per cent.,	average	13.8 per cent.
Resin	9.7 to	11.9	„	10.7

Caraway Seed.—Three samples of the powder yielded respectively 7·67, 6·39 and 6·89 per cent. of ash.

Cascara Sagrada.—Seven parcels assayed for cold water soluble matter gave figures ranging from 21·56 to 26·90 per cent., with an average of 24·04.

Castor Oil.—The examination of some twenty-three samples of medicinal oil has given the following results:—

Specific gravity	0·960 to 0·966
Saponification value	178·5 to 184·5
Refractive index	1·4784 to 1·4803

Catechu.—Two samples of powder, prepared from the B.P. drug, yielded 3·56 and 4·34 per cent. of ash, respectively. A specimen of Black Catechu proved to yield 2·95 per cent. of ash.

Ceresin.—As we have pointed out before, much of the article offered under this name, and more especially in the case of the yellow variety, is a substitute, probably containing colophony. The results obtained for three samples were as follows:—

Specific gravity	0·9985 to 1·003
Melting-point	47·5° to 50°
Saponification value	103·46 to 111·30

Charcoal.—One of the five samples examined contained a proportion of mineral matter in excess of the not too stringent official maximum of 7·5 per cent. The actual figures were:—

6·44, 11·21, 4·64, 2·78, 2·89 per cent. respectively.

Chaulmoogra Oil.—A substitute for true Chaulmoogra Oil (from *Taraktagenos Kurzii*) offered as being obtained from *Hydnocarpus Wightiana* proved to possess characters very similar to those of the genuine oil. The results obtained were as follows:—

Specific gravity at 45°C.	0.9374
Melting point	26°C.
Saponification value	205.8
Iodine absorbed	95.57 per cent.

Cinnamon.—Ash-yield from seven samples of this drug ranged from 2.55 to 3.91 per cent.

Cocoa-butter.—Eleven samples have been examined, none of which showed any departure from the characteristic constants. The actual figures obtained were:—

Melting point	32° to 34°
Saponification value	192.1 to 192.2
Iodine absorbed	36.02 to 39.68 per cent.

Cocoanut Oil.—We find the constants for this oil to fall within narrow limits, little variation being observed. A large number of samples have been examined, giving the following range:—

Specific gravity at 99°C.			
(Water at 15.5°C. = 1.000)	0.8670 to 0.8741
Melting point	25° to 26.5°C.
Saponification value	257.4 to 263.1

Cod Liver Oil.—The following analysis of one consignment of the “A1” Oil of the season 1910-11, from our own factory in the Lofotens, is typical. The figure for free fatty acid is again very low, and is characteristic of a cod liver oil of the very first class. We find the refractive index to fall slightly below the figures usually stated:—

Specific gravity	0.9265
Saponification value	187.7
Iodine absorbed	168.0 per cent.
Unsaponifiable matter	1.11 „
Refractive index	1.4791
Free fatty acid (as oleic acid)	0.26 per cent.
Colour reactions	Normal

Colocynth Pulp.—Three commercial specimens gave the following results:—

	1	2	3
Ash	10.75	6.32	11.62 per cent.
Ether-soluble matter	traces	17.47	4.18 „

A sample of pulp prepared by careful hand picking gave on assay :—

Ash	11.01 per cent.
Ether-soluble matter	2.33 „

Colza Oil.—Several samples have been examined, nearly all of which were satisfactory in character, one sample, however, proved to be highly abnormal in character, and was doubtless adulterated.

	NORMAL OILS.	ABNORMAL OIL.
Specific gravity	0.9145 to	0.9176 0.9246
Saponification value	172.55 to 177.90	185.1
Refractive index	1.4732 to	1.4742 1.4747
Iodine absorbed	—	116.4 %
Unsaponifiable matter	—	1.68 %

Copaiba.—In striking contrast to the results we have had to record in an earlier number of this Report (No. 12 p. 14) not one of the eleven samples of Copaiba tested during the year has shown any indication of the presence of Gurjun oil or balsam.

The quantitative results obtained were as follows :

Specific gravity...	0.9855 to	0.9908	
Resin	54.54	to 67.69%,	averaging 59.8%
Acid value ...	76.33	to 88.43%,	„ 81.2
Ester value ...	4.76	to 16.73%,	„ 9.9
Resin acid factor	0.67	to 0.77%,	„ 0.73

(See Report No. 12.)

Coriander.—Three batches of the powdered seeds yielded 7.15, 7.19 and 5.82 per cent. of ash, respectively.

Cotton-seed Oil.—The two samples examined proved to be quite satisfactory and in each case well-marked characteristic reactions were obtained on the application of both *Halphen* and *Becchi* tests.

Specific gravity	0.9230,	0.9220
Saponification value ...	192.6,	195.0
Iodine absorbed	108.6	— per cent.
Free fatty acid calculated		
as oleic acid	0.16,	0.24 „
Refractive index	1.4732,	1.4730

Cubebs.—For three samples of the powdered drug the figures obtained for ash-yield were:—

4.63, 4.66, and 5.00 per cent. respectively.

Elaterium.—A specimen of English-grown Elaterium gave an analysis:—

Soluble in hot alcohol (90 per cent.) ...	61.0 per cent.
Elaterin	21.4 „ „

Traces of starch were present.

Ergot.—Nine samples were subjected to assay for proportion of water-soluble matter, the range observed being from 14.56 to 20.57 per cent., with an average of 16.7.

Gentian.—Ash was determined on seven batches of the powdered root, the figures obtained ranging from 3.68 to 5.63 per cent. and averaging 4.68 per cent.

Ginger.—The subjoined figures for ash were obtained for powders of various qualities of our own grinding:—

Jamaica	3.64,	3.88 per cent.
African	4.88	„
Cochin	5.60	„

Insect Wax (Chinese Wax).—A single specimen of this somewhat uncommon wax was examined and gave:—

Melting point	80.5°
Saponification value	66.7
Iodine absorbed	3.54 per cent.

Ipecacuanha.—Twenty samples have been assayed during the year for total alkaloid, the process of the U.S.P. being employed for this purpose. Alkaloidal content varied from 1.62 to 2.38 per cent., giving as an average 1.98, a figure distinctly lower than that obtained last year.

Jalap.—A large number of parcels of this drug have been assayed, a rather larger proportion than usual yielding the official percentage of resin. The figures are for the air-dried drugs:—

Total resin...	...	4.48 to 14.32%	average 8.76%
Resin insoluble in ether	...	4.04 to 13.92%	„ 8.40%

The impossibility of obtaining an accurate knowledge of the resin-content of a parcel of Jalap from the assay of a small type sample is shown by our experience in one instance. A bale of the drug purporting to yield about 10 per cent. of resin was sampled, about a pound of the tubers being withdrawn. On drying and grinding, the sample yielded 5.79 per cent. of resin. A second sample, similarly treated, yielded 8.01 per cent., and finally, in order to settle the matter, the whole parcel was ground, mixed and assayed. It proved to yield 9.36 per cent. of resin.

Japan Wax.—Two samples of this wax have been examined, the high iodine figures obtained indicating the presence of the Perilla Oil, now stated to be a constant constituent of the commercial wax.

Specific gravity	—	0·991
Melting point	53°	54°
Saponification value ...	222·45,	209·12	
Iodine absorbed ...	16·16,	15·23	per cent.

Kino.—The figures obtained for three samples show very wide variation in the quality of commercial Kino. Soluble in boiling water:—66·55, 91·00, 82·06 per cent. respectively.

Kousso.—Five samples of the powder obtained from various sources showed evidence of considerable addition of mineral matter. The ash-yield for the five ranged from 16·39 to 21·00 per cent., with an average of 18·42.

Two parcels of the entire drug gave 8·52 and 10·18 per cent. of ash, whilst a sample of the powder ground in our own mills yielded 8·18 per cent.

Linseed Oil.—We have again to record that adulterated linseed oil is not infrequently offered. We give below the results obtained for two such samples:—

	GENUINE OILS		ABNORMAL OILS	
Specific gravity	0·9310 to	0·9340	0·9303,	0·9315
Saponification value	191·6	to 195·6	182·3,	194·5
Iodine absorbed	177·7	to 193·8	172·4,	150·6
Refractive index	1·4805 to	1·4830	1·4819,	1·4778

Mace.—A single sample of true Banda Mace in powder yielded characteristic results when tested:—

Petroleum spirit extract	27·57	per cent.
Ether extract (on the drug after exhaustion with petroleum)	1·86	„
Ash	1·65	„

Male Fern, extract.—We have been able to confirm the truth of the statement of *E. J. Parry* (*P.J.*, 1911 [2] p. 778), to the effect that much of the commercial extract is grossly adulterated. We give below the results obtained by the analysis of six samples on the lines laid down in the above paper, these results clearly showing the non-genuine character of the extracts marked Nos. 1 and 2.

	1	2	3	4	5	6
Specific gravity	0.980	0.9745	1.0231	1.0148	1.0205	1.020
Refractive index at 20° ...	1.4830	1.4840	1.5210(?)	1.5055	1.5065	1.5040
Saponification value ...	204.6	195.1	256.3	235.4	241.0	258.20
Unsaponifiable matter ...	4.94%	3.12%	—	8.60%	93.0%	9.54%
Mean molecular weight of fatty acids	286.1	280.7	—	250.0	247.0	—
Crude Filicin	6.09%	7.16%	—	—	26.04%	28.76%
Behaviour with ten volumes of Petroleum Ether.)	insoluble.	insoluble.	no oil insoluble but much flocculent matter.	as No. 3.	no oil insoluble and little flocculence.	as No. 5.

Myrrh.—The ash obtained from thirteen samples ranged from 3.24 to 9.28 per cent., with an average of 4.69 per cent.

Mustard Oil, expressed.—The figures obtained for the three samples tested show very close agreement:—

Specific gravity	0.921	to	0.924
Saponification value	177.0	to	177.2
Refractive index	1.4720	to	1.4752

Neatsfoot Oil.—The three samples tested during the year proved to be normal in character, giving the low iodine value characteristic of the genuine oil:—

Specific gravity	...	0.9165	to	0.9175
Saponification value	...	195.5	to	197.2
Iodine absorbed	...	70.66	to	71.50 per cent.
Refractive index	...	1.4685	to	1.4690

Nutmeg, Expressed Oil.—The figures given below were obtained from two samples of this fat, and show the very great variation occurring in its characters, especially when compared with those given in our 19th Report:—

Melting-point	38°,	49 5°
Acid value	11·6,	9·8
Saponification value	...	191·2,	200·7	
Iodine absorbed	60·5,	42·2 per cent.

Nut Oil (Arachis Oil).—Five samples have been examined, all proving of satisfactory quality. The figures obtained were:—

Specific gravity...	...	0·9165 to	0·9190
Saponification value	...	191·4	to 194·4
Free fatty acid (as oleic acid)	0·82 to 1·18 per cent.

Olive Oil.—Over a hundred samples of this oil have been submitted to analysis during the year, and we are glad to be able to report that the amount of adulteration experienced has been much less than during recent years.

In our Nineteenth Report (p. 14), we published a comparison of various proposed tests for Arachis oil, showing the *Renard* test (*Archbutt* modification) to be the most reliable. This test we apply to all samples of Olive Oil before passing into stock, and as a result were able to detect the presence of Arachis oil in three casks of one consignment. The proportion was but small, being from the weight of Arachidic Acid (M.Pt. 72·5°) obtained, approximately 8 per cent.

These were the only adulterated oils met with during the year, although considerable difference in general quality naturally occurs with the genuine samples, the percentage of free fatty acid approximately indicating the

grade of the oil. The range of figures obtained for 101 samples was:—

Specific gravity...	...	0.9145 to	0.9180
Free fatty acid (as oleic acid)	0.80 to	13.50 per cent.
Saponification value	...	187.7 to	194.5
Iodine absorbed...	...	80.28 to	86.59 per cent.
Refractive index	...	1.4670 to	1.4718

Opium.—The three parcels assayed for Morphine all showed high percentages, needing large dilution to reduce to the 10 per cent. standard of the official powder. The figures obtained were:—

Morphine (calculated on the dry drug)...	15.87, 15.21, 14.40 per cent.
--	-------------------------------

Palm Oil.—The only sample examined showed the large proportion of free fatty acid usually characteristic of commercial samples. The actual figures obtained were:—

Moisture	3.01 per cent.
Free fatty acid (calculated as palmitic acid)	29.78 „
Saponification value...	203.5

Palm Wax.—A specimen of this wax was received and examined during the year, the results obtained showing its characters to be similar to those of Carnauba Wax.

Specific gravity	0.9655
Melting-point	76°
Saponification value	67.6
Iodine absorbed	10.62 per cent.

Peruvian Balsam.—Four samples have been examined, with results for the most part satisfactory.

Specific gravity	...	1.1468 to	1.1490
Ether residue			
(from 5 grammes)	...	2.60 to	3.09 grammes.
Saponification value of			
Ether residue	...	240.4 to	253.1

Pimento.—Two samples of the powdered drug yielded 3.59 and 3.40 per cent. of ash, respectively.

Poppy-seed Oil.—Two consignments tested during the year proved to correspond to the normal characters of this oil. The figures were:—

Specific gravity	0.9270,	0.9265
Refractive Index	1.4757,	1.4755
Saponification value	...	197.6,	200.0	
Iodine absorbed	132.8,	131.6 per cent.

Quassia.—Ash-yield from three samples of powder ranged from 1.65 to 3.43 per cent.

Resin (Colophony).—Acid values (for three samples) 168.8, 170.4, 159.4.

Rhubarb.—Five samples of ground root proved to yield ash:—6.94 to 8.2 per cent., average 7.54 per cent. A single batch of powdered English Rhubarb gave 6.29 per cent.

Saffron.—During the examination of seven samples the only abnormal feature encountered was an excessive amount of moisture—14.68 per cent.—in one sample. The range observed was:—

Moisture	9.31 to 14.68 per cent.
Ash (on dry Saffron)	...	5.41 to	7.07	„

Scammony.—The figures obtained by the analysis of four samples of reputedly “virgin” Scammony afford interesting reading. They were:—

Ash 21.30, 5.72, 11.32, 2.14 per cent.
 Soluble in ether
 (0.717) ... 38.79, 78.11, 62.78, 76.15 ..

Sesamé Oil.—We have again to report the occurrence of cotton-seed oil in a sample of this oil.

Seal Oil.—A sample of this oil examined gave somewhat unusual results:—

Specific gravity	0.9215
Refractive index	1.4750
Saponification value	188.0
Iodine absorbed	122.3 per cent.

Soya Bean Oil.—A single sample proved to possess the following characteristics:—

Specific gravity	0.9275
Refractive index	1.4775
Saponification value	185.5
Iodine absorbed	135.4 per cent.

Stramonium Leaves.—Two batches of the powder yielded 21.7 and 27.6 per cent. of ash respectively.

Sperm Oil.—Three samples have been examined during the year, two of which were normal in character, No. 3 being adulterated in all probability with a fatty oil. The figures obtained were:—

	1	2	3
Specific gravity	... 0.879,	0.880	0.892
Saponification value	... 133.4,	128.0	140.8
Iodine absorbed	... 81.19,	81.62%	95.8%
Unsaponifiable matter...	35.40,	37.54%	26.50%
Fatty acids	... 63.37,	62.56%	71.61%
Refractive index	... —	—	1.4665

Spermaceti.—We have examined a considerable number of samples during the past year, and after rejecting those which were unsuitable for pharmaceutical use owing to colour, etc., we have obtained the following results for the remaining ten samples:—

Melting-point	44° to 46°
Saponification value	122.5 to 128.6

Storax.—Some years since (Report No. 16, etc.) we called attention to the deterioration in quality of the crude Storax of commerce, an experience which has now been confirmed by *J. C. Umney* (Perf. Record, June, 1911, p. 126). We ourselves find the three samples of the crude drug examined during the present year to show a distinct improvement in quality. The actual figures obtained were:—

Soluble in alcohol (90%)	...	67.78,	62.80,	71.38%
Insoluble in alcohol (90%)	...	2.52,	2.69,	3.77%
Free balsamic acid as benzoic acid	...	1.78,	1.92,	2.50%
Combined balsamic acid as benzoic acid	...	12.79,	13.42,	14.20%

Tolu, Balsam of.—The three samples examined all proved to be of indifferent quality, containing much lower proportions of aromatic esters than those recorded last year.

Soluble in alcohol (90%)	87.50,	82.45,	77.66%
Insoluble in alcohol (90%)	0.82,	1.38,	2.56%
Free balsamic acid as benzoic acid	8.51,	8.95,	7.40%
Combined balsamic acid as benzoic acid	13.62,	16.30,	16.21%

Tragacanth.—A sample of “Indian” Tragacanth yielded 4.86 per cent. of ash, and gave the reactions characteristic of the official drug.

Tung Oil.—A single sample of this rapidly drying Oil gave the following results :—

Specific gravity	0.942
Refractive index	1.5216
Saponification value	192.4
Iodine absorbed	172.2 per cent.

Whale Oil.—A specimen of oil of good quality examined gave :—

Specific gravity	0.925
Refractive index...	1.4760
Saponification value	194.8
Iodine absorbed	112.5 per cent.

ESSENTIAL OILS.

IN this department of analytical work it has unfortunately to be stated that increased knowledge of the constitution of the Essential Oils has been closely followed by more refined and scientific methods of sophistication. Cheap synthetic esters of low molecular weight have been used to cover additions to oils which are dealt with commercially on an ester basis. Acetone has been employed to augment the apparent percentage of aldehyde, while cheap synthetic aldehyde itself has also been used, as in the case of Oil of Cinnamon.

Such methods as these naturally call for much greater vigilance in testing than was necessary when petroleum, alcohol and turpentine formed the chief stock-in-trade of the sophisticator.

Almonds.—We find the refractive index for the synthetic benzaldehyde to be a practically constant figure, 1.5454.

Anise, Star.—This oil has been the source of considerable trouble during the year. In some instances deficiency in anethol was the cause of complaint; in others a diminished solubility in alcohol.

One of the samples belonging to the latter class proved not to dissolve even in five volumes of alcohol (90 per cent.) at ordinary temperatures, the mixture not becoming clear until warmed to 45°C. Other samples, although not forming a clear solution at 15° with three

volumes of alcohol (90 per cent.), did so on warming to about 25°.

The matter has been more fully discussed in an editorial in the *Perfumery and Essential Oil Record* (Dec., 1911, p. 273).

The results of the tests applied to seven samples examined are given *in extenso* below. Nos. 2 and 3 were objected to on account of deficiency in anethol, while Nos. 5, 6 and 7 were the insoluble samples:—

	1	2	3	4	5	6	7
Specific gravity	0.982	0.9755	0.9750	0.9828	0.986	0.988	0.987
Rotation ...	-0.25°	+0.20°	+0.15°	0°	-0.12°	+0.05°	-0.50°
Congeaingpoint	10°	12°	11°	15°	14°	14°	16°
Melting-point ...	16°	13°	14.5°	17°	17°	16°	19°
Refractiveindex	1.5597	1.5491	1.5947	1.5550	1.5510	1.5557	1.5575
With 3 vols. of alcohol (90%)	soluble	soluble	soluble	soluble	not soluble	soluble at 27°	soluble at 21°

Sample No. 5 was further examined, being fractionally distilled, and the density and refractive index of each fraction determined.

	REFRACTIVE INDEX.	SPECIFIC GRAVITY.
1st.—10 per cent.	... 1.5435	... 0.9659
2nd.—15 ,,	... 1.5529	... 0.9814
3rd.—20 ,,	... 1.5570	... 0.9875
4th.—20 ,,	... 1.5590	... 0.9896
5th.—20 ,,	... 1.5597	... 0.9914
Residual.—15 per cent....	1.5406	... 0.9940

The fall in the refractive index of the last portion is very considerable, much more so than is the case with normal soluble Anise Oil.

Bay.—Four samples have been examined during the year, and whilst all were of satisfactory quality, considerable variation in phenol-content was found.

Specific gravity 0.9685 to 0.9855
Phenols 55.7 to 69.7 per cent.
Refractive index	... 1.5240 to 1.5202

Bergamot.—None of the three samples tested dissolved clearly in two volumes of alcohol (80 per cent.). The figures obtained were as follows :

Specific gravity	0·8875,	0·8825,	0·8820
Esters as linalyl acetate	40·26,	37·97,	39·44%
Residue at 100°	5·76,	4·57,	4·73%
Refractive index	1·4675,	1·4678,	1·4671
Refractive index of residue after evaporating 90 per cent.	1·5073	—	—

Cajeput.—Four samples were examined, the results obtained being quite normal:—

Specific gravity 0·9180 to 0·9220
Refractive index 1·4695 to 1·4710

Caraway.—We append the results obtained by examining six samples of foreign oil, the last two of which were of somewhat poor quality:—

Specific gravity	0·9175	0·9185	0·9160	0·9120	0·9100	0·9090
Rotation	+77·2°	+77·1°	+74·75°	+77·87°	+78·0°	+78·5°
Distillate above 200° ...	53%	61%	—	57%	49%	50%
Refractive index	1·4891	1·4890	1·4878	1·4860	1·4893	1·4875

Chamomile.—Two parcels of English oil examined gave very similar results:—

Specific gravity 0·9185,	0·9180
Refractive index 1·4449,	1·4440

Here again we note that the Specific gravity in each case is higher than the maximum limit proposed by the above authors.

Cinnamon.—We again find that samples of this oil from abroad show higher percentages of Cinnamic aldehyde than those claimed by English distillers to be characteristic of the genuine oil. For five samples the range obtained was:—

Specific gravity	1·028 to 1·035
Cinnamic aldehyde	72·55 to 77·83 per cent.
Refractive index	1·5893 to 1·5950

Citronella.—Both of the samples examined proved to contain a satisfactory proportion of alcohols:—

Specific gravity	...	0.901,	0.9089
Rotation	...	−11.10°,	−11.0°
Geraniol content	...	68.28,	69.81 per cent.

Coriander.—The only sample examined proved to possess normal characters.

Specific gravity	0.873
Rotation	+10.35°
Refractive index	1.4696
Soluble in 3 vols. of alcohol (70 per cent.)				

Cumin.—We report upon samples of both English and foreign oils. In physical properties there appears to be very little difference between them.

	ENGLISH.		FOREIGN.	
Specific gravity	0.9115,	0.9145	0.9145,	0.9215
Rotation	+4.70°,	+4.75°	+4.25°,	+4.32°
Refractive index	1.5003,	1.5000	1.5020,	1.5023

Dill.—Last year we drew attention to the too-narrow limits for specific gravity recommended by *Hill and Umney* for inclusion in the new Pharmacopœia, we wish again to express the hope that the figures now official, viz., 0.905 to 0.920, will be allowed to stand. Our results for the year obtained on four samples were:—

Specific gravity	0.9095 to	0.9175
Rotation	+70.25°	to +75°
Refractive index	1.4850 to	1.4876

Clove.—A considerable number of samples have been assayed for Eugenol and tested in other ways. In no case was any deviation from the normal characters observed.

Specific gravity	1.052	to 1.058
Eugenol	80	to 90 per cent.
Refractive index	1.5305	to 1.5340

Copaiba.—A sample of the bulked distillate from several hundred pounds of Copaiba gave the following results :—

Specific gravity	0.9030
Rotation	-14.13°
Refractive index	1.4971

Distilled between 250° and 263°

Eucalyptus.—The following figures for a number of samples offered commercially as B.P. show considerable differences, more especially with respect to optical rotation.

Specific gravity	0.9110 to	0.9215
Rotation	-9.59° to	0°
Refractive index	1.4610 to	1.4673

Globulus oils gave :—

Specific gravity	0.921 to	0.9265
Rotation	-2.2° to	+1.07°
Refractive index	1.4610 to	1.4630

Eucalyptol determined on one sample by the phosphoric acid method, amounted to 72 per cent.

Fennel.—Five samples of this oil have been submitted to our analysts during the year, the results being uniformly satisfactory.

Specific gravity	0.9675 to	0.9740
Rotation	+13.06° to	+14.68°
Congeeing point	+° to	6°
Refractive index	1.5343 to	1.5363

Geranium.—Two samples of African Rose Geranium Oil gave the following data :—

Specific gravity	0.901, 0.8965
Alcohols as geraniol (free and combined)	71.27, 68.83 per cent.
Refractive index	1.4965, 1.4688

Ginger.—A single sample was examined and furnished normal results.

Specific gravity	0.8845
Rotation	-33.2°
Refractive index	1.4930

Juniper.—As a means of distinguishing the genuine berry oil, we continue to apply the test proposed by *Umney*, which consists in observing the refractive index of the residue remaining after distilling off 80 per cent. of the oil. This constant exhibits well-marked differences, as the following figures show, when comparing the genuine oil with the substitutes described as Juniper Wood Oils.

	JUNIPER OILS.		A TYPICAL "WOOD" OIL.
Specific gravity	...	0.8675 to 0.8720	0.8795
Rotation	...	-7.0° to -12.33°	-23.87°
Refractive index	...	1.4775 to 1.4825	1.4775
"Residual" refrac- tive index	...	1.5023 to 1.5055	1.4891

Lavender.—We have met with two instances during the year in which foreign lavender oil, guaranteed to be genuine, would be excluded by the minimum ester limit of 30 per cent., which has been proposed as an official standard. For ten samples the results obtained were:—

Specific gravity	...	0.8883 to 0.8995
Esters as linalyl acetate	26.69	to 35.31 per cent.
Refractive index	...	1.4620 to 1.4652

Lemon.—Considerable trouble has been experienced during the year with regard to this oil, many samples met with being deficient in citral, and in some cases giving undoubted evidence of sophistication. We give below the results obtained in the examination of

a series of Lemon Oils, the citral in each case being determined by *Bennett's* hydroxylamine method. It is obvious that many of these samples would be rejected by the application of the not unreasonably high standard of 3·5 per cent.

Specific gravity	0·8592	0·8584	0·8572	0·8580	0·8570	0·8567	0·8590	0·8580	0·8575	0·8575
Rotation ...	48·25°	57·12°	60·20°	60·28°	62·0°	58·2°	58·67°	56·76°	60·50°	59·90°
Rotation of first fraction ...	41·25° [†]	54·80° [‡]	—	—	—	57·8° [†]	58·61° [*]	57·09° [*]	—	—
Refractive index	1·4741	1·4740	1·4755	—	1·4750	1·4745	—	1·4760	1·4745	1·4750
Citral... ..	2·32 [†] %	2·19%	4·05 [†] %	3·70 [†] %	2·75 [†] %	3·35 [†] %	2·92 [†] %	4·10 [†] %	2·89 [†] %	2·96%

† Twenty per cent., possessing an unmistakeable odour of turpentine.

* Fifteen per cent.

‡ Twenty-five per cent.

Lemon-Grass.—One sample only was examined which proved to be fairly rich in aldehyde, but somewhat low in density. A test for Acetone was made by fractional distillation, with negative results.

Specific gravity	0·887
Citral	79·7 per cent.
Refractive index	1·4858

Matico.—A single sample tested proved to give satisfactory figures.

Specific gravity	0·9725
Refractive index	1·5075

Nutmeg.—Two specimens of the volatile oil have been examined, the results obtained falling within the usually accepted limits.

Specific gravity...	0·8960,	0·9065
Rotation	+16·25°,	+21·30°
Refractive index	1·4795,	1·4831

Orange.—We almost invariably find a considerable difference in the physical characters of the bitter and sweet orange oils, this being especially marked with the

optical rotation. We append figures relating to both varieties:—

	BITTER		SWEET.
Specific gravity ...	0·8545,	0·8530	0·8510
Rotation ...	+91·75°,	+89·75°	+99·2°
Refractive index...	1·4770,	1·4749	1·4728

Pennyroyal (European).—One sample only was examined, yielding normal results:—

Specific gravity	0·9395
Rotation	+22·5°
Refractive index	1·4850
Soluble in 2 vols. of alcohol (70 per cent.).				

Peppermint.—Oils of American and Japanese origin have been examined, the figures obtained being given below. Those for the Japanese oil refer of course to the partially dementholised oil:—

	JAPANESE.		AMERICAN.	
Specific gravity ...	0·901	to 0·903	0·906	to 0·913
Menthyl acetate ...	7·67	to 10·30%	9·13	to 10·92%
Free Menthol ...	46·60	to 53·40%	56·24	to 63·02%
Refractive index ...	1·4605	to 1·4622	1·4610	to 1·4640

Petitgrain.—Satisfactory percentages of ester have to be recorded in the case of the two samples examined, the complete figures being:—

Specific gravity	0·8895,	0·8875
Esters as linalyl acetate...	43·48,	41·67	per cent.
Refractive index...	...	1·4655,	1·4641

Both samples formed a clear solution with 2 volumes of alcohol (80 per cent.).

Pimento.—The characters of the only sample examined proved to be quite satisfactory.

Specific gravity...	1·050
Eugenol	78 per cent.
Refractive index	1·5337

Pine (*Pinus pumilio*).—The two samples assayed showed somewhat marked variation in the proportion of esters present.

Specific gravity...	...	0·8695,	0·8705
Rotation...	...	-7·75°,	-6·05°
Esters as bornyl acetate	7·43,	4·02 per cent.	
Refractive index	...	1·4787	

Pine (*Pinus Sibirica*).—We note that both samples examined showed Bornyl Acetate in excess of the minimum standard recommended. Assuming the ester content to be the therapeutically valuable portion of this oil, there appears to be good reason for the substitution of this oil for the variety at present official. The figures obtained for two samples were as follows:—

Specific gravity...	...	0·9135,	0·9145
Rotation...	...	-41·8°,	-42·0°
Esters as bornyl acetate	31·94,	36·52 per cent.	
Refractive index	...	1·4720,	1·4701

Rosemary.—Results obtained during the year enable us to confirm the statement that under the monograph proposed for inclusion in the forthcoming Pharmacopœia some genuine English distillates would be excluded by the non-admission of dextro-rotatory oils. Our Laboratory Journal shows the following figures:—

	ENGLISH.	FOREIGN.		
Specific gravity	0·902	0·905,	0·910
Rotation	-2·87°	+3·10°,	+1·88°
Refractive index...	...	1·4680	1·4710,	1·4719
Solubility in 2 volumes of alcohol (90 per cent.)	clear	clear	clear	clear

Rue.—We have not for some considerable time met with a sample of the grossly-adulterated oil formerly common. The only specimen examined gave:—

Specific gravity	0.8375
Rotation	-1° (about)
Congeaing point	8°
Soluble in 3 volumes of alcohol (70 per cent.)	

Sandal Wood.—No unusual features are presented by the results recorded during the year. We still find that a temperature of 20° to 25° is necessary to form a clear solution with 6 volumes of alcohol (70 per cent.). The range observed for the six samples examined was :—

Specific gravity	0.9735 to 0.9785
Rotation	-16.05° to -17.75°
Alcohol as Santalol	89.90 to 95.96%
Refractive index	1.5030 to 1.5045

Savin.—We have again to note that foreign distilled oils frequently vary considerably from the usually accepted standards. Two samples examined gave :—

Specific gravity	0.912, 0.913
Rotation	+52.50°, +47.45°
Distillate below 200°	48, 54 per cent.
Saponification value	121.9, 102.6
Refractive index	1.4761, 1.4778

Sassafras.—The physical characters of the six samples examined showed but little variation :—

Specific gravity	1.069 to 1.079
Rotation	+2.75° to +3.86°
Refractive index	1.5284 to 1.5310

Thyme.—The only sample of genuine oil of Thyme examined gave :—

Specific gravity	0.920
Phenols	37.3 per cent.
Rotation	-0.80°

Turpentine.—We have before commented on the great variation in optical rotation experienced recently in this oil, and the figures of the year show this to be still more pronounced. The range observed for many samples of American oil was :—

Specific gravity	0·866	to	0·870
Rotation	-10·50°	to	+12·62°
Refractive index	1·4702	to	1·4730

Fractionation and other tests proved satisfactory in all cases.

Wormseed (American).—The two samples examined were of satisfactory character and gave closely agreeing results.

Specific gravity	0·974,	0·975
Rotation	-5·75°,	-6·08°
Refractive index	1·4778,	1·4766

CHEMICAL PREPARATIONS.

THE following records show that constant watchfulness is necessary to preserve the high standard of purity we have always set ourselves in this class of pharmaceutical preparations. A large number of the chemicals used in dispensing we manufacture ourselves, and these, equally with purchased supplies, are rigidly scrutinised before being passed into stock.

Arsenic and lead as impurities continue to claim a large share of our attention in this section, and we may say that in what follows, quantitative references to the former are expressed in terms of the element Arsenic (As).

Ammonium Carbonate.—We have obtained very satisfactory consignments of this substance during the year, samples when perfectly freed from effloresced matter, titrating as high as 99·7 per cent. of the official salt.

Ammonium Persulphate.—Samples of this salt are frequently offered containing much sulphate. Assayed by titrating the amount of iodine liberated from Potassium Iodide in presence of a catalyst ($\text{CuSO}_4 + \text{FeSO}_4$) from 91·5 to 95·9 per cent. of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ was the range obtained.

Amyl Acetate.—We find commercial samples to fall into two classes, one containing 70 to 75 per cent. of ester, the other 90 to 97 per cent.

Antimony Sulphide (black).—A sample of the crude native sulphide proved to contain but 4.02 per cent. of siliceous matter, but was considerably contaminated with Arsenic, the proportion present being approximately 0.35 per cent.

Bole, Armenian.—Since this substance is occasionally used as a colouring matter for food-stuffs, we always examine for Arsenic. One sample only contained an excessive proportion, viz., 50 parts per million. In the remaining five the amount present varied from 0 to 5 parts per million.

Bismuth Salts.—We have experienced some difficulty in obtaining both the carbonate and sub-nitrate sufficiently free from chlorides for our purposes.

Borax.—Last year we commented upon the improved quality of this substance with respect to arsenical contamination. We regret to note that our experience during the latter part of the year has been such that we have had considerable difficulty in procuring supplies sufficiently free from Arsenic to satisfy our requirements.

We are given to understand that this difficulty is largely due to a change in the source of much of the native boron compound imported.

Forty samples have been examined, and we have found the amount of Arsenic present to range from 0 to 160 parts per million, the latter being of course exceptionally large. We consider that, as a fair standard, Borax sold for pharmaceutical purposes should not contain more than 4 parts per million of Arsenic.

Boric Acid.—We find Boric Acid to remain commendably free from Arsenic, but have had occasion

to object to samples containing an excessive proportion of sulphates.

Calcium Carbonate.—A sample of “*Calcii Carbonas Præcipitatus*” drawn from our ordinary stock proved to possess the following composition:—

Calcium Carbonate (CaCO_3)	...	99.13	per cent.
Ferric Oxide and Alumina ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$)	0.28	„
Magnesia (MgO)	0.29	„
Chlorine (Cl)	0.02	„
Siliceous matter	0.01	„
Sulphates and Phosphates	...	very small traces.	

Calcium Phosphate.—Arsenical contamination has been met with in this substance, 20 parts per million having been recorded in one instance.

Chloroform.—We have found it necessary during the year to reject a consignment of Chloroform on account of its failure to comply with the tests of the Pharmacopœia, a dark brown colour being produced on shaking with sulphuric acid.

Chromic Acid.—Many grades of this substance are offered commercially, during the year we have met with samples assaying from 33.7 to 100.0 per cent. of the trioxide (CrO_3).

Citric Acid.—This acid has been remarkably free from metallic contamination for many years, but we were disagreeably surprised during the past year to meet with a sample containing no less than 80 per parts per million of lead. We were assured by the makers that the contamination was accidental, but the very fact emphasises the necessity for constant watchfulness in this respect.

For the remaining samples, lead in no case exceeded 10 parts per million while arsenic was usually absent or

at most 1 to 2 per million. Ash-yield was very low ranging from 0.008 to 0.022 per cent.

Iron Arsenate.—One of the four samples examined proved to be deficient in ferrous arsenate, titration indicating but 5.42 per cent. of the anhydrous compound. The other samples contained 19.73, 18.19, and 18.45 per cent., respectively.

Iron, Reduced.—Two samples tested gave fairly satisfactory results:—

Metallic Iron	95.0, 89.9 per cent.
Arsenic	20, 40 parts per million

Iron Sulphate.—As a result of the year's experience we have to repeat our statement of last year's Report as to the difficulty in obtaining 99.4 per cent. of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in crystal form. For fine green crystals of our own manufacture we obtained 97.5 per cent.

Glycerin.—Arsenic in one sample of foreign re-distilled glycerin amounted to 20 parts per million, a proportion seldom now met with in this grade.

Hydrogen Peroxide.—Many samples met with have proved to contain an amount of non-volatile residue in excess of the maximum official limit, figures as high as 0.71 per cent. have been obtained in some instances.

Magnesia.—Some difficulty is experienced in obtaining the heavy variety sufficiently free from sulphates to comply with the somewhat vaguely worded official test.

Magnesium Carbonate.—A similar difficulty appears to prevail here, one sample examined proved to contain as much as 0.98 per cent. of sulphate

(SO₄). Another sample contained 18 parts of Lead per million, and we have also met with a parcel in which quite a considerable proportion of Arsenic was present.

Magnesium Sulphate.—Many samples of this salt have been offered containing an excessive proportion of chlorides, in three cases where the amount was determined the figures were 0·27, 0·43, 0·12 per cent. (MgCl₂).

Mercuric Oxide, Red and Yellow.—We have had to reject a considerable number of parcels of both oxides on account of excessive non-volatile residue. For the Red oxide seven such samples gave from 0·19 to 0·43 per cent., satisfactory specimens yielding as low as 0·002 per cent.

One parcel of precipitated oxide contained 0·60 per cent. non-volatile matter.

Pilocarpine Nitrate.—One only of the samples examined gave a satisfactory melting-point, viz., 172·5°, the results obtained for the others ranged from 165° to 170°.

Potash, Sulphurated.—One parcel tested proved of unsatisfactory quality, containing but 38 per cent. soluble in alcohol.

Potassium Binoxalate.—Seven samples of this compound have been examined, five of which were considerably contaminated with Lead.

We have made some experiments with a view to establishing the composition of the commercial compound sold under this designation.

Five samples, procured through the ordinary channels, have been examined. Potassium in each was

determined as sulphate (K_2SO_4), and acidity by titration with standard alkali. The results obtained are expressed in the following table, the acidity being given in terms of cc. of normal alkali required to neutralize one gramme of the salt.

	Potas- sium (K)	Acidity	$\begin{matrix} \text{COOH, COOH} \\ \text{COOH, COOK} \end{matrix} 2H_2O$ Calculated from K	$\begin{matrix} \text{COOH, COOH} \\ \text{COOH, COOK} \end{matrix} 2H_2O$ Calculated from acidity
No. 1	15.39%	11.95	100.23%	101.17%
No. 2	15.32%	11.9	99.78%	100.74%
No. 3	---	11.75	---	99.48%
No. 4	14.37%	10.8	---	---
No. 5	14.29%	10.7	---	---

The above results show that the first three samples corresponded fairly accurately to Potassium Quadroxalate, $\begin{matrix} \text{COOH, COOH} \\ \text{COOH, COOK} \end{matrix} 2H_2O$. Samples No. 4 and 5 contained less potassium than is necessary for the quadroxalate, and are at the same time less acid than this salt. The constitution of these latter is doubtful, and unfortunately the quantity of material at our disposal did not allow of further work being done.

Potassium Oxalate (neutral).—Lead occurring in appreciable quantity has been found as an impurity in a sample of this salt.

Potassium Bitartrate.—All samples examined proved to be of highly satisfactory quality. By direct titration the amount of $KHC_4H_4O_6$ present amounted to practically 100 per cent. in all cases. Bitartrate, determined according to our invariable practice, by titration of the soluble alkalinity of the incinerated substance, gave somewhat lower figures, the percentages found ranging from 98.36 to 99.87 per cent. We find that the lower figures occur when sulphate is present, and

are presumably due to loss of soluble carbonate by interaction with calcium sulphate.

Lead in no case exceeded 3 parts per million, and for Arsenic $1\frac{1}{2}$ parts per million was the highest figure obtained.

Soap, Hard.—We have again met with soaps offered as Sapo Durus P.B., which analysis shows to be prepared from fats other than Olive Oil. The following are the results obtained during the year:—

	B.P. SOAP.		OTHER SAMPLES.	
Mean molecular weight of fatty acids	284·8,	291·5	252·2,	252·4
Iodine absorbed by fatty acids ...	82·28,	84·50%	67·63,	57·01%

Sulphur.—Out of twenty-eight samples of Precipitated Sulphur examined, but six were passably free from Arsenic, the amount in one instance reaching the high figure of 80 parts per million.

Sublimed Sulphur, we find, rarely contains much Arsenic, the highest figure registered being 6 parts per million, while not one of ten samples of Roll sulphur tested showed more than 1 per million.

Tartaric Acid.—In our experience the purity of samples of this acid has been generally satisfactory. Twenty samples have been examined, the highest figure for Lead obtained being 17 parts per million, while the average figure for the whole was 8 parts. Arsenic was usually absent, although 2 parts per million was recorded in one instance.

Mineral matter in this substance still gives some trouble, the official limit of 0·05 per cent. being decidedly stringent. Samples examined have yielded figures for ash ranging from 0·025 to 0·26 per cent., the average result being 0·08 per cent.

GALENICAL PREPARATIONS.

WE do not doubt that in the forthcoming revised Pharmacopœia greater prominence will be given to the subject of the standardisation of Galenicals, and we believe that the many contributions to this subject from these laboratories, published in these reports and elsewhere, will be found of value in fixing efficient and practicable standards. We again revert to the much-discussed subject of Spiritus Ætheris Nitrosi, and may say that through our experiments generally we are so fully convinced that loss of Ethyl Nitrite is practically entirely due to volatilisation that we have designed an apparatus by which we fill the spirit direct from the containers, in which it is prepared, into the bottles in which it is despatched, without any exposure to air.

In this way we ensure, as far as possible, that the pharmacist receives the spirit at the strength at which it leaves our laboratory, a strength which is so adjusted as to leave a very substantial margin above the B.P. standard.

Asafetida, Tincture of.—We experience considerable difficulty in the preparation of this tincture using the official menstruum. With the better-class samples of Asafetida there is a pronounced tendency for the tincture to separate into layers, the upper containing oil and resin, the lower more or less gummy. The difficulty is easily overcome by the use of alcohol of a little higher strength, and we would suggest that the use of alcohol (90 per cent.) or a mixture of equal parts of this and

alcohol (70 per cent.) should be made official in the next Pharmacopœia.

Colchicum, Extract of.—The bulked extract of this year's manufacture assayed, has yielded as 1·76 per cent. of total alkaloids, a figure higher than the average for this preparation.

Conium, Extract of.—A normal result was obtained for the season's extract, a sample yielding 0·041 per cent. of alkaloidal hydrochlorides.

Hydrastis, Liquid Extract.—A batch of this preparation manufactured on the large scale yielded 1·78 per cent. of Hydrastine, a result somewhat lower than those previously obtained.

Hyoscyamus, Green Extract of.—A sample from the bulked extract prepared from some tons of herb yielded 0·082 per cent. of total alkaloids by titration, a result nearly identical with that obtained last year but low in comparison with the average of a number of years.

Jaborandi, Liquid Extract of.—A sample prepared from the leaves of *Pilocarpus microphyllus* yielded 0·53 per cent. of alkaloids on assay, and we trust that in the next edition of the British Pharmacopœia this variety of Jaborandi will replace that at present official.

This latter variety (*P. Jaborandi*) is practically unobtainable commercially, and we have shown that the substitute (*P. pennatifolius*) usually supplied is of very low alkaloidal strength.

(See Report No. 14, p. 13)

Podophyllin.—A sample of a large batch prepared in our laboratories from the rhizome of *Podophyllum peltatum* showed:—

Ash	0.60 per cent.
Insoluble in alcohol (90 per cent.)	1.36 ..
Insoluble in Ammonia	4.08 ..

Rhubarb, Compound Powder of.—The following figures have been obtained for samples from batches prepared in our Laboratory on the large scale:—

Ash	68.94,	67.94,	68.19 per cent.
Magnesia (MgO)...	—	64.86,	— ..

Strophanthus, Tincture.—We note that *Haycock* (*P.J.*, 1911 [2] p. 553) has confirmed the accuracy and convenience of the method for the assay of this Tincture published by the late Mr. John Barclay (*P.J.*, 1896 [2] p. 463).

We are unable however to agree with the standard (0.1 per cent. w/v) for the tincture put forward by the above writer. For the tincture of the *Pharmacopœia* of 1885 in which one ounce per pint of the seed was used, the experiments carried out in 1896 showed an average of about 0.37 per cent. We believed at the time of the issue of the new B.P. in 1898, that the reduction in strength of the tincture to one-half did not give a corresponding reduction in glucosidal strength, the altered method of preparation apparently producing an effect in this direction (*P.J.*, 1898 [2] p. 655).

More extended experience however showed us that a figure of 0.2 per cent. w/v more correctly represented the average strength of the new tincture, this strength being strictly proportional to that of the 1885 preparation. This figure was accordingly incorporated in the list of proposed standards, published in our Report, No. 11, of 1903.

Sweet Spirit of Nitre.—This preparation has been so frequently the subject of notes in these

Reports that we feel we owe some apology to our readers for again alluding to it.

The series of experiments we record here was put in hand in order to obtain further information on the subject of the cause of loss of Ethyl Nitrite on keeping.

From a batch of the spirit, recently prepared, some two gallons was withdrawn into a large bottle and assayed, from this bulk eleven one-pint stoppered bottles were filled, some of the bottles being of deep amber colour, others of the usual pale-green.

Immediately after filling one of the bottles was assayed, and to six of the remainder small quantities of water, and in one instance glycerine, were added as shown in the table appended, the whole ten being stored either on a light shelf or in a dark cupboard for a period of ten months at ordinary room temperature.

At the end of this time the bottles were opened and the contents immediately assayed. The results obtained are tabulated below and are corrected to eliminate the dilution effect caused by the addition of the water or glycerine.

Description	Character of bottle	Condition of storage	Cubic centimetres of Nitric Oxide at 0° and 760 mm. from 5 c.c. of the spirit.	Sp. Gr.
Original Spirit	—	—	32·63	0·8381
.. immediately after bottling	—	—	31·61	—
Undiluted Spirit after ten months ..	Green	Light	30·48	0·8389
.. .. .	Amber	Light	30·42	0·8389
.. .. .	Green	Dark	32·00	0·8388
.. .. .	Amber	Dark	32·00	0·8389
pirit + 1% of water after ten months	Amber	Dark	31·21	0·8408
.. + 2%	Green	Light	31·73	0·8426
.. + 2%	Amber	Light	31·35	0·8427
.. + 2%	Amber	Dark	30·54	0·8431
.. + 5%	Amber	Dark	30·45	0·8491
.. + 5% of glycerin ..	Amber	Dark	32·14	0·8522

There are obvious inconsistencies in these results, for example, the undiluted spirit after storing for ten months is apparently stronger in ethyl nitrite than when first bottled. This apparent anomaly we are, however, inclined to attribute to variation in the amount of exposure during bottling, and to differences in the stoppering of the bottles.

On the whole, the results are such as to establish several facts. The chief is that the loss by hydrolysis in a spirit of the official alcoholic strength is, practically speaking, nil during a period of ten months, and that while small quantities of water do produce an appreciable effect, that effect is very small, and is inconsiderable in comparison with the loss which we have previously shown, may be sustained by volatilisation if the spirit is exposed to air. (*See* Report 17-18, p. 36).

Minor points arising are that 5 per cent. of glycerine exercises some preservative effect, and that a loss of approximately 3 per cent. may occur in the mere act of transferring from one bottle to another.

In the early part of the year, wishing to gain some information as to the strength of Sweet Spirit of Nitre as retailed in Birmingham pharmacies, we purchased ounce quantities from various quarters of the city. These purchases were made over the counter, as a matter of every-day retail business, and we think the results afforded by the assay testify, on the whole, to the care taken by local pharmacists in the preservation and sale of this article. The percentages of Ethyl Nitrite by weight, shown on analysis, were as follows:—

1	2	3	4	5	6
1·82	2·76	1·94	2·04	2·74	2·12

(B.P. minimum standard is 1·75 per cent.).

TABLE

SHOWING SUGGESTED STANDARDS, RANGES
OF SPECIFIC GRAVITY, ETC.,
FOR
GALENICAL PREPARATIONS.

Name of Preparation.	Range of Specific Gravity.	STANDARD. (Where there is no active principle mentioned the figure given represents total extractive.) Grammes per 100 c.c.	Range of percentage (by volume of Alcohol.	REMARKS.
Acetum Cantharidis	1.066 to 1.072	3.62 HA.	11 to 12.5	
" Ipecacuanhæ	0.991 to 0.993	0.1 total alkaloid		
" Scillæ	1.035 to 1.040	8.0		
Collodium	0.775 to 0.782	4.0 acetic acid		
" Flexile	0.790 to 0.797	2.0		
Decoctum Aloes Compositum	1.001 to 1.004	6.5	16 to 17	
" Granati Corticis Conc.		5.6		
" Hæmatoxyli Conc.		30.0		
Extractum Belladonnæ Liquidum	0.896 to 0.912	3.0	72.0 to 75.0	
" " Viride		*0.75 total alkaloid		
" Cascaræ Sagradæ Liquidum	1.070 to 1.080	1.0 total alkaloid		
" Cimicifugæ Liquidum	0.890 to 0.900	26.0	17.0 to 18.0	
" Cinchonæ Liquidum	1.115 to 1.150	12.5	74 to 78	
" Cocæ Liquidum	1.004 to 1.014	*5.0 total alkaloid	10.5 to 12.0	
" Colocyntidis Compositum†		0.5 total alkaloid	45.5 to 49.5	
" Ergotæ Liquidum	1.020 to 1.027	20 ether soluble		
" Filicis Liquidum	1.000 to 1.019	15.0	31.5 to 32.5	
" Glycyrrhizæ Liquidum	1.140 to 1.150	42.0	16.0 to 18.0	
" Hamamelidis Liquidum	1.025 to 1.050	21.0	33 to 34	
" Hydrastis Liquidum	1.025 to 1.040	2.0 hydrastine	37.5 to 38.5	
" Hyoscyami Viride		0.1 total alkaloid		
" Ipecacuanhæ Liquidum	0.885 to 0.910	*2.00 to 2.25 total alkaloid	75.0 to 79	
" Laborandi Liquidum	1.020 to 1.040	20.0	33 to 34	
" Jalapæ		20 resin		
" Nucis Vomizæ Liquidum	0.950 to 0.970	*1.5 strychnine	57.5 to 61.5	
" Opii Liquidum	0.985 to 0.990	*0.75 morphine	17.5 to 18.5	

Extractum Pareiræ Liquidum	..	1'050 to 1'065	22'5	20 to 22
Sarsæ Liquidum	1'080 to 1'090	20'0 (without glycerine)	15 to 17
Strophanthi †	4'0 strophanthin	16 to 20
Taraxaci Liquidum	..	1'070 to 1'090	25'0	
Glycerinum Acidi Borici	..	1'345		
" " Carbolicæ	..	1'230		
" " Tannici	..	1'288 to 1'292		
" Aluminis	1'288 to 1'292		
" Boracis	1'280 to 1'295		
" Pepsini	1'190 to 1'200		
Infusum Aurantii Conc.	(1 fluid dr. should dissolve 12,000 grains hard boiled white of egg.	
" " Compositum Conc.	10'2	
" Buchu Conc...	7'2	
" Calumbæ Conc.	6'0	
" Caryophylli Conc.	3'5	
" Cascarillæ Conc.	5'0	
" Chirataæ Conc.	2'0	
" Cinchonæ Acidum Conc.	4'5	
" Cuspariæ Conc.	1'0 total alkaloid	
" Digitalis Conc.	8'0	
" Gentianæ Compositum Conc.	1'6	
" Krameriæ Conc.	6'0	
" Lupuli Conc.	8'0	
" Quassiaæ Conc.	7'0	
" Rhei Conc.	0'25	
" Scoparii Conc.	10'0	
" Senegæ Conc.	15'0	
" Sennæ Conc...	10'0	
" Serpentariæ Conc.	14'0	
" Uvæ Ursi Conc.	4'5	
Linimentum Aconiti	0'865 to 0'875	10'0	78'0 to 80'0
" " Belladonnæ	..	0'880 to 0'900	0'25 total ether soluble alkaloid	69'0 to 72'0
			* 0'375 total alkaloid	

* Officially Standardized.

† Scaunmony resin

‡ P. J., 1898 (2), p. 665.

Name of Preparation.	Range of Specific Gravity.	STANDARD. (Where there is no active principle mentioned the figure given represents total extractive.)		Range of per-centage (by volume) of Alcohol.	REMARKS
		Grammes per 100 c.c.			
Linimentum Camphoræ	0.924 to 0.927	21.5 camphor +		57.0 to 58	
" " Ammoniatum	0.866 to 0.872			62 to 64.0	
" " Saponis	0.895 to 0.900			19.0 to 21.0	
Liquor Calumbæ Conc.	0.990 to 0.996	4.25		18.0 to 19.0	
" Chiratzæ Conc.	1.000 to 1.010	5.5		18.0 to 19.0	
" Cusparæ Conc.	1.005 to 1.015	10.0		16.0 to 18.0	
" Hamamelidis	0.980 to 0.985			76.0 to 77.0	
" Iodi Fortis	1.010 to 1.025	11.5 iodine		18.0 to 19.0	
" Krameriæ Conc.	1.015 to 1.025	10.0			
" Picis Carbonis	0.855 to 0.865	2.5		18.0 to 19.0	
" Quassiazæ Conc.	0.975 to 0.980	0.30		18.0 to 19.0	
" " Rhei Conc.	1.020 to 1.030	12.5		18.0 to 20.0	
" Sarsæ Compositus Conc.	1.030 to 1.040	15.0		18.5 to 22.0	
" Senegæ Conc.	1.015 to 1.025	12.5		18.0 to 19.0	
" Sennæ Conc.	1.040 to 1.060	17.5		18.0 to 19.0	
" Serpentariæ Conc.	0.990 to 1.000	5.0		14.4 to 16.6	
Mistura Sennæ Co.	1.110 to 1.118	16.5			
Oxymel	1.320*	4.4 acetic acid			
" " Scillæ	1.320*	2.0 acetic acid			
Pulv. Glycyrrhizæ Comp.		9.0 sulphur			
" Rhei Comp.		68 to 70 ash			
Succus Belladonnæ	0.980 to 0.990			18.0 to 22.0	
" Conii	0.980 to 0.990			18.0 to 22.0	
" Hyoscyami	0.980 to 0.990			18.0 to 22.0	
" Scoparii	0.980 to 0.990			18.0 to 22.0	
" Taraxaci	0.995 to 1.000				
Syrupus Aromaticus	1.148 to 1.156				
" Aurantii	1.270 to 1.280				
" Calcii Lactophosphatis	1.310 to 1.320				
" " Cascariæ Aromaticus	1.110 to 1.125				

Syrupus Chloral	1'315 to 1'320	
Codeinæ	1'320 to 1'328	
" Ferri Phosphatis	1'270	
" Phos. c. Quin. et Strych.	1'275 to 1'285	
" Glucosi	1'363 to 1'370	
" Hemidesmi	1'325 to 1'330	
" Limonis	1'290 to 1'300	
" Pruni Virginianæ	1'300 to 1'320	
" Khei	1'310 to 1'320	
" Rosæ	1'325 to 1'330	
" Scillæ	1'330 to 1'335	
" Sennæ	1'280 to 1'290	
" Tolutanus	1'330 to 1'335	
" Zingiberis	1'300 to 1'310	
" Tinct. Aconiti	0'890 to 0'897	
" Aloes	0'970 to 0'985	
" Arnicæ	0'890 to 0'894	
" Asafetidæ	0'914 to 0'920	
" Aurantii	0'880 to 0'890	
" Belladonnæ	0'912 to 0'920	
" Benzoini Comp.	0'900 to 0'905	
" Buchu	0'930 to 0'935	
" Calumbæ	0'920 to 0'925	
" Camphoræ Comp.	0'915 to 0'920	
" Cannabis Indicæ	0'844 to 0'850	
" Cantharidis	0'835 to 0'838	
" Capsici	0'890 to 0'897	
" Cardamomi Comp.	0'945 to 0'950	
" Cascarillæ	0'900 to 0'905	
" Catechu	0'972 to 0'978	
" Chirataë	0'920 to 0'924	
" Chloroformi et Morph. Comp.	1'010 to 1'020	

[each fluid dr.
*Fe₃ (PO₄)₂—1 gr. in

0'02 total ether
soluble alkaloid

8'5
0'6

10'0

2'0

*0'05 total alkaloid

About 5'0 total balsamic acids,
of which about 2'0 are free
and 3'0 combined.

4'0

1'0

*0'05 morphine

4'0

0'25

1'50

6'50

1'6 resin

14'5

0'8

68'0 to 70'0

38'0 to 41'0

68'8 to 69'8

56'0 to 62'0

72'5 to 76'0

60'0 to 61'0

68'5 to 72'5

56'0 to 57'0

56'0 to 58'0

56'0 to 59'0

83'0 to 86'0

87'0 to 89'0

67'5 to 69'5

54'0 to 56'0

62'5 to 67'5

51'0 to 53'0

56'0 to 58'0

52'0 to 54'0

+ Grammes per 100 grammes.

* Officially Standardized.

Name of Preparation.	Range of Specific Gravity.	STANDARD.		REMARKS.
		(Where there is no active principle mentioned the figure given represents total extractive.)		
			Grammes per 100 c.c.	Range of percentage (by volume) of Alcohol.
Tinct. Cimicifugæ	2.0	57.0 to 59.0
" Cinchonæ	* 1.0 total alkaloid	63.0 to 64.5
" Cinchonæ Comp.	5.00	62.0 to 68.0
" Cinnamomi	* 0.5 total alkaloid	66.0 to 68.0
" Cocci	2.4	42.0 to 44.0
" Colchici Seminum	2.5	42.0 to 44.0
" Conii	0.10 total alkaloid	66.0 to 68.5
" Croci	0.09 total alkaloid	56.0 to 58.0
" Cubebæ	3.00	83.0 to 85.0
" Digitalis	2.0 oleo-resin	53.0 to 57.5
" Ergotæ Ammon.	3.6	48.0 to 52.0
" Ferri Perchlor	4.0	18.0 to 21.0
" Gelsemii	0.025 total alkaloid	57.0 to 59.0
" Gent. Comp.	5.0	42.5 to 43.5
" Guaiaci Ammon.	15.0	69.0 to 71.0
" Hamamelidis	2.0	42.0 to 44.0
" Hydrastis	2.5	56.0 to 58.0
" Hyoscyami	0.008 total alkaloid	40.5 to 42.5
" Iodi	* 2.5 iodine	84.0 to 86.0
" Jaborandi	0.048 total alkaloid	42.0 to 44.0
" Jalapæ	* 1.5 resin	65.0 to 67.0
" Kino	5.0 kino-tannic acid	43.0 to 44.5
" Krameriæ	5.0	56.0 to 58.0
" Lavand Comp.	0.6	86.5 to 88.5
" Limonis	2.0	67.5 to 71.5
" Lobeliæ Æther.	0.07 lobeline	58.0 to 60.0
" Lupuli	4.0	54.0 to 57.0
" Myrrhæ	5.6	84.0 to 86.0
" Nucis Vomicaæ	* 0.25 strychnine	58.0 to 64.0

Tinct. Opii	0.955 to 0.960	0.75 morphine	40.0 to 44.0
" Opii Ammon.	0.895 to 0.900	*0.113 morphine	60.0 to 63.0
" Podophylli	0.850 to 0.855	3.65 resin	84.5 to 89.0
" Pruni Virg.	0.930 to 0.938	3.0	52.0 to 57.0
" Pyrethri	0.895 to 0.900	2.25	66.0 to 69.0
<hr/>						
" Quassia	0.944 to 0.948	0.05 quassin	42.0 to 44.0
" Quillaia	0.918 to 0.924	1.25	56.0 to 58.0
" Quininæ	0.885 to 0.895		72.5 to 76.0
" " Ammon.	0.925 to 0.928		53.0 to 54.0
" Rhei Comp.	0.970 to 0.975	4.50 (Exclusive of Glycerine)	51.0 to 53.0
" Scilla	0.955 to 0.962	10.0	52.0 to 54.0
" Senega	0.935 to 0.938	6.5	55.0 to 57.0
" Sennæ Comp.	0.985 to 0.994	10.00	38.5 to 40.5
" Serpentaria	0.896 to 0.900	2.00	66.0 to 68.0
" Stramonii	0.950 to 0.964	0.04 total alkaloid	42.0 to 44.0
" Strophanthi	0.892 to 0.895	0.2 strophanthin	67.0 to 69.0
" Sumbul	0.895 to 0.900	2.5	66.0 to 69.0
" Tolutana..	0.860 to 0.865	about 3 balsamic acids; 3 of which are free	80.0 to 82.0
<hr/>						
" Valeriana Ammon.	0.935 to 0.942	3.0	50.0 to 54.0
" Zingiberis	0.835 to 0.840	0.4	88.5 to 89.5
Vin. Colchici	1.010 to 1.015		

* Officially Standardized

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